Glass and Ceramics, Vol. 71, Nos. 1 – 2, May, 2014 (Russian Original, Nos. 1 – 2, January – February, 2014)

SCIENCE FOR CERAMIC PRODUCTION

UDC 666.76

FRACTURE SURFACE MICROSTRUCTURE OF ELEMENTARY ALUMINUM OXIDE FILAMENTS

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Translated from *Steklo i Keramika*, No. 2, pp. 13 – 15, February, 2014.

The surface microstructure of a fracture of an elementary aluminum-oxide filament and the radial structural nonuniformity of an elementary aluminum oxide filament were studied. The thickness of the strong outer shell and the structure of a weaker core of the elementary filament were determined. It was proposed on the basis of tensile rupture tests of elementary filaments with different diameters that the outer shell thickness affects the strength of elementary aluminum oxide filaments.

Key words: elementary aluminum oxide filaments, scanning electron microscopy, microstructure of fracture.

The aluminum oxide filaments developed at the All-Russia Scientific Research Institute of Aviation Materials (VIAM) have a high potential of finding applications in technical articles in many sectors of domestic industry [1]. Complex investigations of the properties and structure of filaments are needed in order to realize this potential.

One time-consuming form of research is the study of the structure of elementary filaments and fibers at the macro and micro levels.

It is shown in [2, 3] that fibers and elementary filaments possess structural nonuniformity in the radial direction. This nonuniformity is manifested in the presence of an outer shell, whose structure and physical-mechanical properties are different from those of the core, on the surface of fibers and elementary filaments.

The aim of the present work is to investigate the structural nonuniformity along the radius of elementary aluminum oxide filaments by means of optical microscopy and the microstructure of the fracture surface by means of x-ray microscopy.

The investigation of the radial nonuniformity of elementary filaments 12 – 14 μm in diameter was conducted in transmitted light under an Opton (Germany) optical microscope. Before the investigation the elementary filaments were placed in an immersion liquid with $n = 1.515$ and covered with standard cover glass. Photomicrographs were made 1 h and 6 h after the preparation was assembled (Figs. 1 and 2, respectively).

The photomicrograph in Fig. 2 clearly shows that the immersion liquid permeates the elementary filament while the regions directly adjoining the outer shell are not permeated. This phenomenon can be explained by the presence of a dense outer shell with thickness equal to 8 – 12% of the diameter of the filament and a core with significant open porosity; in addition, the pore size and pore distribution over the transverse section vary considerably from one sample to another.

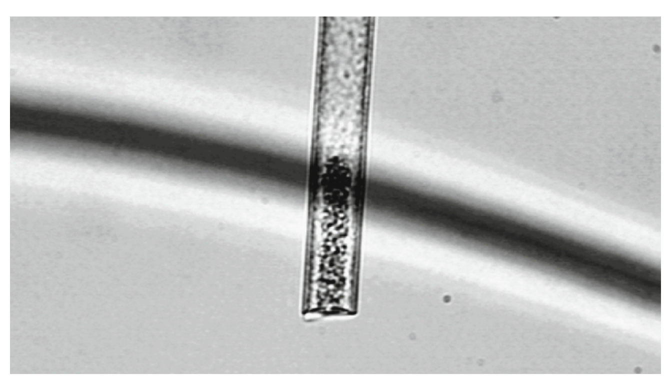

Fig. 1. Permeation with an immersion liquid; permeation time 1 h.

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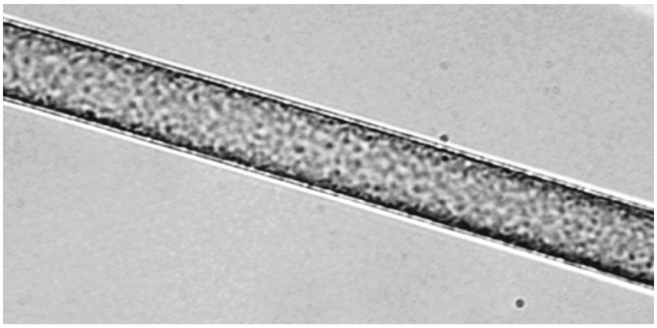

Fig. 2. Permeation with an immersion liquid; permeation time 6 h.

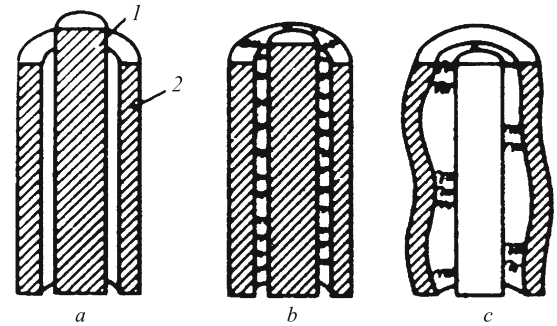

Fig. 4. Physical model of an elementary filament: *a*) complete absence of bonding; *b*) high bonding strength; *c*) weak bonding.

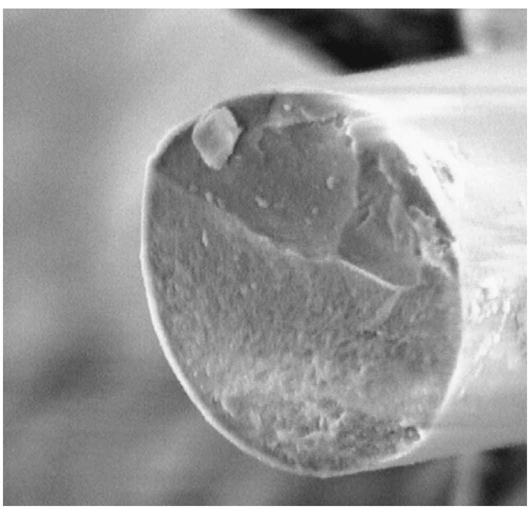

Fig. 3. Outer shell and core of elementary filament.

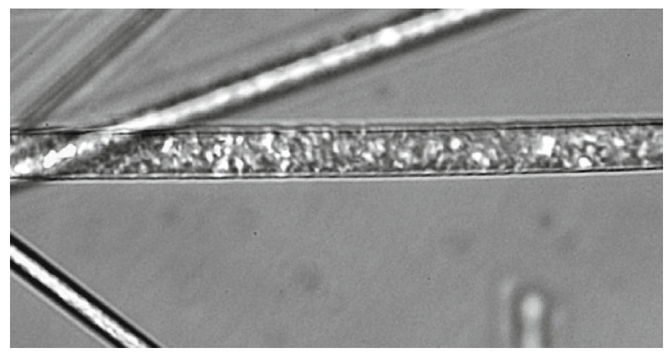

Fig. 5. Bonding between the outer shell and core of a filament.

To obtain data on the microstructure of the fracture surface of elementary filaments with high resolution elementary aluminum oxide filaments were studied by means of scanning electron microscopy (SEM).

Structural analysis of samples of elementary aluminum oxide filaments was performed by SEM in a DSM-960 Opton (Germany) electron microscope with accelerating voltage 5 kV and maximum spatial resolution about 100 nm (for conducting samples). The image of the surface was formed by secondary electrons with accelerating voltage 5 kV.

The radial structural nonuniformity of an elementary aluminum oxide filament is clearly visible in the photomicrograph displayed in Fig. 3. The thickness of the outer shell ranges from 4 to 6% of the diameter of the elementary filament. The discrepancy in the estimate of the outer shell thickness by means of optical and x-ray microscopy is due to the specific nature of the experimental methods.

As a rule, a two-layer cylinder is used as the physical model of a fiber and an elementary filament (Fig. 4) [4]. The outer shell is denser and its properties differ from those of the core. Depending on the strength of the bonding between the outer shell and the core of the elementary filament four variants of the interaction between them are possible: complete absence of bonding, high bonding strength, weak bonding and a combination of the second and third variants.

An examination of the images (Figs. 3 and 5) shows that the variant with high bonding strength between the outer shell and core of the elementary filament is realized in elementary aluminum oxide filaments, and in some cases there is local weakening of this bonding because of the presence of density fluctuations in the core.

Here

$$\sigma_{sh} > \sigma_c, \tag{1}$$

where σ_{sh} is the strength of the elementary filament; σ_c is the strength of the core of the elementary filament;

$$\varepsilon_{sh} > \varepsilon_c, \tag{2}$$

where ε_{sh} is the rupture elongation of the outer shell of the elementary filament and ε_c is the rupture elongation of the core of the elementary filament.

Judging from the form of the fracture surface (see Fig. 3) it can be supposed that when an elementary filament is stretched to rupture the more brittle core fractures first, then the stronger and more plastic outer shell fractures and the edge of the outer shell becomes rounded after rupture.

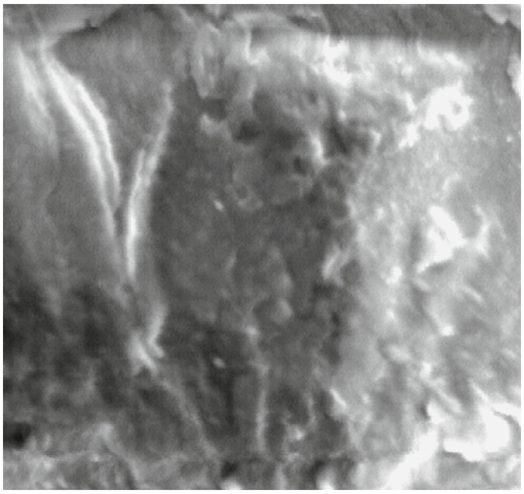

Fig. 6. Layered structure of the core of an elementary filament; × 10,000.

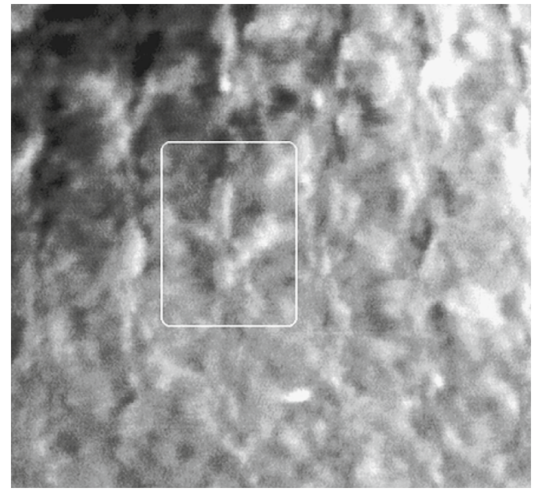

Fig. 7. Intergrown grains of the core of an elementary filament; × 20,000.

Photomicrographs of the fracture of samples of an elementary filament under magnification 10,000 and 20,000 are presented in Figs. 6 and 7, respectively. The core of the elementary aluminum oxide filament is nonuniform in the axial direction and has a layered structure (see Fig. 6). A grainy structure of the core of an elementary filament can be seen in the photomicrograph (see Fig. 7). There is a large size variance (from 100 × 200 to 300 × 700 nm) from one sample to another and the geometric shape varies (circular and oval). The kernel consists mainly of groups of intergrown grains. A structural element comprising four intergrown grains (two 200 × 400 nm grains and two 250 × 600 nm grains) stands out in Fig. 7.

It is shown in [3, 5] that the strength of a fiber depends on the outer shell thickness. No dependence of the outer shell thickness on the diameter of the experimental elementary filaments was found. It can be supposed that there is no such dependence for the elementary fibers studied.

In summary, the larger the diameter of the elementary filaments, the smaller the ratio of the area of the outer shell to the area of the core is. Correspondingly, the smaller the diameter, the stronger the elementary filaments are. This is confirmed by tension to rupture tests performed on elementary filaments. The results of the tests can be grouped as a function of the strength and diameter of an elementary filament as follows [6]:

– from 1100 to 1700 MPa for diameters from 8 to 10 μm;
– from 800 to 900 MPa for diameters from 11 to 14 μm;
– from 350 to 550 MPa for diameters from 15 to 24 μm.

This could be associated with the facts that the stronger outer shell has the same thickness for all elementary filaments and as the diameter increases the ratio of the areas of the outer shell and core decreases.

CONCLUSIONS

1. Elementary aluminum oxide filaments are structurally nonuniform in the radial direction.
2. The strength and rupture elongation of the outer shell of an elementary aluminum oxide filament are higher than those of its core.
3. The thickness of the outer shell of an elementary filament equals 5% of the diameter of the filament and is more uniform than the core.
4. The core of the elementary filament is characterized by a grain structure with open porosity. The grains show a large size variance and differ in geometric shape.

We thank Maksimov Vyacheslav Gennad'evich for assistance in the preparation of this article.

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